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December 29, 1959

ROHM & HAAS COMPANY

REDSTONE ARSENAL RESEARCH DIVISION

HUNTSVILLE, ALABAMA



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Report No. S-23

USE OF A NEW PROPELLANT COMPOSITION
IN THE PERSHING MISSILE

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Use of a New Propellant Composition in the
Pershing Missile.

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UNCLASSIFIED REPORT

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GUIDED MISSILES, PROPELLANT GRAINS, ROCKET PROPULSION,
SURFACE TO SURFACE
IDENTIFIERS: PERSHING

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ROHM & HAAS COMPANY

REDSTONE ARSENAL RESEARCH DIVISION
HUNTSVILLE, ALABAMA

REPORT NO. S-23

USE OF A NEW PROPELLANT COMPOSITION IN THE PERSHING MISSILE

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December 29, 1959

ARMY ORDNANCE CORPS
Project Number TU2-10
RESEARCH ON ROCKET PROPELLANTS AND ROCKET MOTORS
Contract No. DA 01-021-ORD5135

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INTRODUCTION

In contract modification number 20 to contract DA-01-021 ORD-5135, the Redstone Arsenal Research Division of Rohm & Haas Company was requested to study the feasibility of petrin acrylate propellants for the Pershing system. The evaluation study was to be carried out in ten weeks and was to include several petrin acrylate compositions. The Pershing system requires a composition having a comparatively low burning rate and a high specific impulse.

SUMMARY AND CONCLUSIONS

A number of compositional changes were made to a standard petrin acrylate composition in an effort to decrease the burning rate while maintaining a high specific impulse. None of the changes produced a composition having a burning rate low enough to be used with a star perforation. One of the compositions developed had a burning rate low enough to be used with a slotted-tube perforation and this composition was tested in small-scale motors (L/D ratio was the same as that of the Pershing but diameter was 6-inches as compared to 39.82 for the full-scale motor). Pressure-time traces obtained from these tests were quite satisfactory.

Propellant processing studies were carried far enough to allow casting of satisfactory motors for ballistic evaluation. Considerable additional work would be required to develop satisfactory processing techniques for the production of large motors.

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PROPELLANT COMPOSITION STUDIES

A propellant having a burning rate of approximately 0.28 in./sec. at 600 psi is required for the Pershing if a star-shaped perforation is to be used. In addition, the specific impulse at 600 psi must be maintained at a high value ($F_{1000}^0 = 245 \text{ lbf-sec/lbm}$).

Previous burning rate studies made with petrin acrylate propellant indicated that even with unground ammonium perchlorate the burning rate of composition QZ had a minimum value of 0.41 in./sec. at 600 psi. The later addition of magnesium oxide as a coating on the ammonium perchlorate to improve thermal stability and processing increased the burning rate to 0.45 in./sec. Since the specific impulse of QZ composition was 248 lbf-sec/lbm compositional modifications seemed to be possible without reducing impulse below 245 lbf-sec/lbm.

Several approaches to the problem of decreased burning rates were available, including the use of oxidizers other than ammonium perchlorate, coated oxidizers, additives, cooled binder systems, different copolymers, and decreased solids content. In addition, there was a possibility that burning rates higher than 0.28 in./sec. could be tolerated if the web thickness were increased.

Although a burning rate depressant would be desirable because the small amounts which would be added would have a negligible effect on other propellant characteristics and propellant processing, a number of additives had been evaluated previously, and none had a significant effect. This approach was not pursued further.

Because the large oxygen content of the binder in petrin acrylate compositions reduces the requirement for oxygen from the oxidizer there seemed to be a good possibility that lower burning rates could be obtained

by substituting other oxidizers for a portion of the ammonium perchlorate in QZ composition. Two oxidizers, RDX (trimethylenetrinitramine) and ammonium nitrate were examined.

Replacement of up to three-fourths of the ammonium perchlorate by RDX

	<u>RDX-2</u>	<u>RDX-4</u>
Ammonium perchlorate	28.20	15.20
Petrin acrylate	14.70	14.70
Triethylene glycol dinitrate	17.50	17.50
Ethyl hexyl acrylate	1.80	1.60
Polyester P-920	0.50	0.70
Aluminum	8.00	8.00
RDX	28.30	42.30

did not decrease the impulse significantly; compositions fired in 2C4¹ motors gave impulses near 240 lbf-sec/lbm. which is essentially the value obtained with QZ composition in this motor size. Moreover, burning rates were lowered to .26 in./sec. at 600 psi., well within the requirements for a star geometry.

However, shock sensitivity tests showed that composition RDX-2 had a minimum diameter for detonation between 3/16-in. and 1/4-in., while QZ_{bn}, the composition used in the Zeus, has a minimum diameter between 2-in. and 2.5-in. The card gap sensitivity test showed this composition to be about 80% as sensitive as composition C-4 booster material and about 70% as sensitive as pressed tetryl. Since the shock sensitivity of the composition was more characteristic of a high explosive than of a propellant, further work was terminated.

¹2C4 refers to a static test motor 2-inches ID, 4-inches long with a cylindrical perforation having a diameter of 1 or 1.5 inches.

The replacement of up to 15% of the ammonium perchlorate by ammonium nitrate

	4K515 and <u>E-42</u>
Ammonium perchlorate	46.80
Petrin acrylate	14.50
Triethylene glycol dinitrate	17.40
Ethyl hexyl acrylate	2.50
Polyester P-920	0.80
Aluminum	8.00
Ammonium nitrate	10.00

also decreased the burning rate. When 10% NH_4NO_3 was used, the rate was decreased to about .38 in./sec., while 15% NH_4NO_3 decreased the rate to less than .3 in./sec. However, physical properties suffered, and the hygroscopicity problem would require facility changes before large motor castings could be made. Specific impulses of the NH_4NO_3 -containing compositions were approximately 236 lbf-sec./lbm., when determined in 2C4 motors and 240 lbf-sec./lbm. when determined¹ in 6C11.4 static test motors.

Work at NOTS showed that large reductions in the burning rates of Nitrasol propellant could be obtained by coating ammonium perchlorate with silanes and fluorocarbon polymers. Kel-F oil was tested and produced little change in the burning rate. A coating of methyltriethoxysilane reduced the burning rate to .4 in./sec., but the physical properties of the propellant were very poor. When MgO-coated ammonium perchlorate was coated with methyltriethoxysilane the propellant did not cure. A copolymer of 80/20 heptafluorobutyl acrylate/acrylic acid gave only a slight reduction in burning rate, and increased the impact sensitivity of the ammonium

¹6C11.4 motors indicate a static test motor 6-inches ID, 11.4-inches long with a cylindrical grain perforation having a diameter of 5 inches.

perchlorate; the 50% fire level of the coated ammonium perchlorate was 18-inches (1Kg weight) as compared with more than 32-inches for uncoated perchlorate. A similar sensitivity increase was found by NOTS.

Copolymers other than ethylhexyl acrylate were tested for their effect on burning rate. A series of methacrylates (lauryl, decyl-octyl, and stearyl) produced no change in rate, and a fluorocarbon type, C₇ fluoro-acrylylcarbamate, considerably increased the burning rate.

Since the perchlorate decomposition is probably the limiting reaction in the combustion of petrin acrylate propellant, a reduction in burning rate seemed possible by reducing the perchlorate concentration. This effect had previously been observed in a plastisol double-base system. Compositions having the lowest perchlorate content had the lowest burning rates. The effect of reduced perchlorate concentration on the burning rate was tested with a composition containing 40% ammonium perchlorate and 20% aluminum.

	<u>E-58</u>
Ammonium perchlorate	40.00
Petrin Acrylate	17.40
Triethylene glycol dinitrate	19.90
Ethyl hexyl acrylate	1.90
Polyester P-920	0.60
Aluminum	20.00
Ethyl centralite	0.20

The burning rate was reduced to approximately 0.4 in./sec. at 600 psi, but the F_{1000}^0 was also reduced (240 lbf-sec./lbm.) and the density of the composition was low (.061 lbm/cu.in.).

Since the burning rate of QZ propellant had been increased when

magnesium oxide was added as a coating for the ammonium perchlorate a magnesium stearate-magnesium oxide coating was tested in an effort to decrease rates. Burning rates were decreased by this coating, and there was only a small change in impulse. However, the impact sensitivity of ammonium perchlorate was increased considerably by the addition of magnesium stearate, thereby increasing propellant processing hazards.

Since the high energy of the binder seemed to prevent lower burning rates an attempt was made to decrease the energy of the binder by increasing the copolymer concentration. When the ethylhexyl acrylate concentration was increased from 1.6% to 6%

	<u>BCP-4</u>
Ammonium perchlorate	55.0
Petrin acrylate	13.92
Triethylene glycol dinitrate	15.88
Ethyl hexyl acrylate	6.00
Polyester P-920	1.00
Aluminum	8.00
Ethyl centralite	0.20

the burning rate was reduced to 0.33 in./sec. at 600 psi and F_{1000}^0 measured at 1000 psi in 2C4 motors was greater than 235 lbf-sec./lbm. However, when a composition containing 5.5% ethyl hexyl acrylate

	<u>E-29</u>
Ammonium perchlorate	47.80
Petrin acrylate	10.50
triethylene glycol dinitrate	16.15
Ethyl hexyl acrylate	5.50
Polyester P-920	1.00
Aluminum	18.00
Ethyl centralite	0.20

was tested in 6C11.4 standard test motors it was found that impulse decreased rapidly as pressure decreased; at pressures near 400 psi F_{1000}^0 was less than 220 lbf-sec./lbm. Further investigation showed that even 2.3% ethyl hexyl acrylate

	<u>E-90</u>
Ammonium perchlorate	52.85
Petrin Acrylate	14.20
Triethylene glycol dinitrate	17.00
Ethyl hexyl acrylate	2.30
Polyester P-920	0.45
Aluminum	13.00
Ethyl centralite	0.20

would lower the impulse at low pressures, even though the impulse remained high when charges were fired at 1000 psi. As a result of these findings, the maximum copolymer concentration was fixed at 1.6%.

Low impulses obtained at low pressures seemed to indicate that aluminum was not being burned efficiently. The effect of higher ammonium perchlorate concentrations was therefore tested in an effort to improve combustion efficiency. Static test results indicated that combustion efficiency decreased¹ as oxidizer concentration was increased.

Since burning rates were increased as oxidizer concentration increased the aluminum concentration was fixed at approximately 18%; compositional studies made earlier had shown that an impulse plateau existed near this point. Studies on a double-base plastisol system had also indicated that a maximum impulse was obtained with an aluminum concentration near 18%.

¹The study of the effect of aluminum concentration was complicated by the use of a fairly high concentration of flake aluminum which was used to thicken the propellant during processing. Flake aluminum is known to give less efficient combustion than atomized aluminum.

The most effective means of reducing burning rate without adversely affecting impulse, density, or physical properties was found to be an additive originally evaluated in the plastisol system. This was calcium gluconate which decreased the temperature coefficient and burning rate without decreasing impulse. When 2% calcium gluconate was added to a QZ composition the burning rate was decreased more than 10% without reducing the impulse. A series of 6C11.4 motor firings indicated that a QZ composition containing 2% calcium gluconate added had a burning rate of 0.42 in./sec. at 600 psi with an impulse near 244 which was nearly pressure independent.

	<u>QZ-970</u>
Ammonium perchlorate	47.85
Petrin acrylate	14.90
Triethylene glycol dinitrate	17.00
Ethyl hexyl acrylate	1.60
Polyester P-920	0.45
Aluminum	18.00
Ethyl centralite	0.20
Calcium gluconate	2% added

In short, 2% calcium gluconate decreased burning rates 7% without affecting physical properties or impulse. Two other pairs of compositions

	<u>E-48</u>	<u>E-68</u>
Ammonium perchlorate	52.85	52.85
Petrin acrylate	14.90	14.90
Triethylene glycol dinitrate	17.00	17.00
Ethyl hexyl acrylate	1.60	1.60
Polyester P-920	0.45	0.45
Aluminum	13.00	13.00
Ethyl centralite	0.20	0.20
Calcium gluconate		2% added

substantiated these findings.

Although the reduced burning rates of modified QZ compositions were still higher than the 0.28 in./sec. desired for use in a star-perforated grain a burning rate of 0.41 in./sec. at 600 psi could be used with a slotted tube grain geometry. This composition was designated RM.

	<u>RM</u>
Ammonium perchlorate	44.00
Petrin acrylate	14.70
Triethylene glycol dinitrate	17.00
Ethyl hexyl acrylate	1.60
Polyester P-920	0.50
Aluminum	20.00
Calcium gluconate	2.0

QZ composition was modified by replacing 2% of the ammonium perchlorate with aluminum and 2% of the ammonium perchlorate with calcium gluconate to give the required burning rate. The combustion products of this composition should be CO, Al_2O_3 , and H_2/H_2O in a ratio of about 4/1 which is close to optimum for a highly aluminized system.

First tests with composition RM indicated that the burning rate was exactly 0.41 in./sec. at 600 psi, which was the desired figure. However, the propellant was somewhat inferior and specific impulse was less than 240 lbf-sec./lbm. As the quality of the propellant improved the impulse increased to about 242 lbf-sec./lbm. and the burning rate fluctuated between 0.41 - 0.44 in./sec. RM composition appears to be quite suitable for use in the first stage of the Pershing.

Ballistic test results of the compositions tested in 6C11.4 standard test motors are summarized in Table I. In general, two

motors were tested from each batch. The values of "n" are good to one place only. All firings were made at + 70° F., because of time and money limitations.

TABLE I

Ballistic Test Results of Compositions Tested in 6C11.4 Standard Test Motors

Designation	Composition	r_{600}	n	F_{1000}^0 @ Pressure (psi)		
QZ-963-964	QZ with bu grind ammonium perchlorate	.45	.41	243 @ 360	241 @ 550	244 @ 850
QZ-970-974	QZ _{bu} with 2% calcium gluconate added	.42	.32	244 @ 450	242 @ 610	246 @ 1000
E-48-54	QZ _{bu} with 1.3% aluminum (+ 5% ammonium perchlorate)	.47	.32	235 @ 380	242 @ 550	246 @ 1000
E-55-57	E-48 with 3.86% ethyl hexyl acrylate (-1.3% petrin acrylate)	.37	.34	217 @ 300	227 @ 450	237 @ 685
E-58-60	60% solids, 20% aluminum	.36	.25	2 shots only	241 @ 500	241 @ 1090
E-61-63	E-48 + cross-linker	.47	.39	239 @ 400	244 @ 650	246 @ 1050
E-64-66	QZ with 5% aluminum (+ 13% ammonium perchlorate)	.49	.41	231 @ 400	238 @ 570	246 @ 1000
E-67-73 (Odd Nos.)	E-48 with 1% magnesium stearate, 1% magnesium oxide or ammonium perchlorate	.43	.28	237 @ 380	243 @ 520	246 @ 850
E-68-74 (Even Nos.)	E-67 with 2% calcium gluconate added	.41	.37	242 @ 415	243 @ 600	246 @ 1040
E-75-80	E-48 with 2% calcium gluconate replacing ammonium perchlorate	.44	.27	241 @ 500	241 @ 650	244 @ 1050
E-81-83	E-58 + Cross-linker	.40	.32	240 @ 390	240 @ 600	245 @ 1550
E-84-85, 109	E-55 -3% ammonium perchlorate, -2% triethylene glycol dinitrate, +5% aluminum	.37	.30	230 @ 400	232 @ 560	243 @ 900
E-87, 88, 89	E-55 +5% aluminum, with 3% ethyl hexyl acrylate	.39	.34	231 @ 420	242 @ 670	248 @ 1030
E-90-92	E-55 with 2.3% ethyl hexyl acrylate	.45	.31	234 @ 375	240 @ 520	248 @ 930
E-93-95	E-55 +5% aluminum, +.5% ethyl hexyl acrylate	.35	.28	225 @ 365	231 @ 550	242 @ 800
E-96-98	E-55	.37	.25		236 @ 580	247 @ 1250
E-100, 101, 108	E-48	.47	.37	241 @ 400		246 @ 1080
E-102-104	E-58 with 2.7% ethyl hexyl acrylate + .9 P-920 replacing triethylene glycol dinitrate	.36	.35	230 @ 420	232 @ 660	240 @ 1000
E-105-107	QZ with 3% ethyl hexyl acrylate replacing triethylene glycol dinitrate (140 aluminum)	.42	.45	233 @ 420	242 @ 640	243 @ 870

TABLE I (cont'd)

Ballistic Test Results of Compositions Tested in 6C11.4 Standard Test Motors

Designation	Composition	r_{600}	n	F_{1000}^0 @ Pressure (psi)		
E-110-113	QZ with 2% aluminum + 2% calcium gluconate replacing ammonium perchlorate	.41	.30	235 @ 500	238 @ 700	239 @ 850
E-118-120	E-110	.42	.35	240 @ 450	241 @ 640	244 @ 1050
E-26-28, 40, 41	BCP-2	.28	.35	218 @ 400	232 @ 600	
E-29, 30	QZ with 5.5% ethyl hexyl acrylate, replacing petrin acrylate	.32	.35	222 @ 400	230 @ 500	
E-31, 32	QZ with 5.5% ethyl hexyl acrylate, replacing triethylene glycol dinitrate	.32	.35	228 @ 400	238 @ 600	
E-33, 34	QZ with 5.5% ethyl hexyl acrylate, replacing petrin acrylate, ammonium perchlorate and aluminum; triethylene glycol dinitrate increased	.30	.32	222 @ 400	233 @ 600	
E-35, 36	E-33 with triethylene glycol dinitrate replacing petrin acrylate	.34	.40	226 @ 450	238 @ 520	2 shots only
E-37-39	50% solids with 2% ethyl hexyl acrylate and QZ $\frac{M-1}{TEGDN}$ and $\frac{APC}{Al}$ ratios	.41	.35	230 @ 530	233 @ 650	237 @ 1000
E-42-44	QZ with 10% ammonium nitrate, replacing aluminum, 2.5% ethyl hexyl acrylate replacing ammonium perchlorate	.38	.35		240 @ 650	241 @ 900
E-45-47	BCP-4	.34	.35	218 @ 370	233 @ 600	

GRAIN CONFIGURATION DESIGN

Three types of grain configuration were considered for the Pershing first stage. The wagonwheel was discarded immediately because the thickest web obtainable was approximately $1/7$ of the motor diameter (D) requiring a burning rate much too slow for petrin acrylate compositions.

The internal burning star was a second possibility. A web of around $0.28 D$ appeared possible but this design would require a burning rate of about 0.30 in./sec. , which is not obtained easily with petrin acrylate compositions.

The third possibility was the slotted tube, a design in which the web is proportional to loading fraction. Since all previous analyses of the slotted tube configuration had been based on an assumption that the grain was a right circular cylinder extensive design modifications were required to compensate for the elliptical head and tail of the Pershing motor. An empirical approach to the problem seemed to be most promising.

A 6-in. diameter motor which was somewhat shorter than a scale Pershing was used for initial tests. Charges having a cylindrical perforation were cast in these motors and slots having various lengths were cut by hand. Fourteen charges were fired in the short motor and a reasonably good trace had been obtained by the time a scale motor became available. The scale motor had an ID of 6-in., and a length of 12-in.; the cylindrical perforation had a diameter of 1.2 inches, giving a web of 2.4 in. and a loading fraction of over 90%. There is no sliver in the slotted tube configuration.

Eight scale Pershing motors were cast. Because of processing difficulties encountered with the new composition being tested, two of the motors could not be fired. Four motors gave pressure-time traces which indicated that the charges did not burn normally; propellant bond failure was noted at the tail end of the motors before they were fired. One trace obtained when the two good motors were fired approached the shape desired (Fig. 1). The regressivity could be corrected by decreasing the slot length.

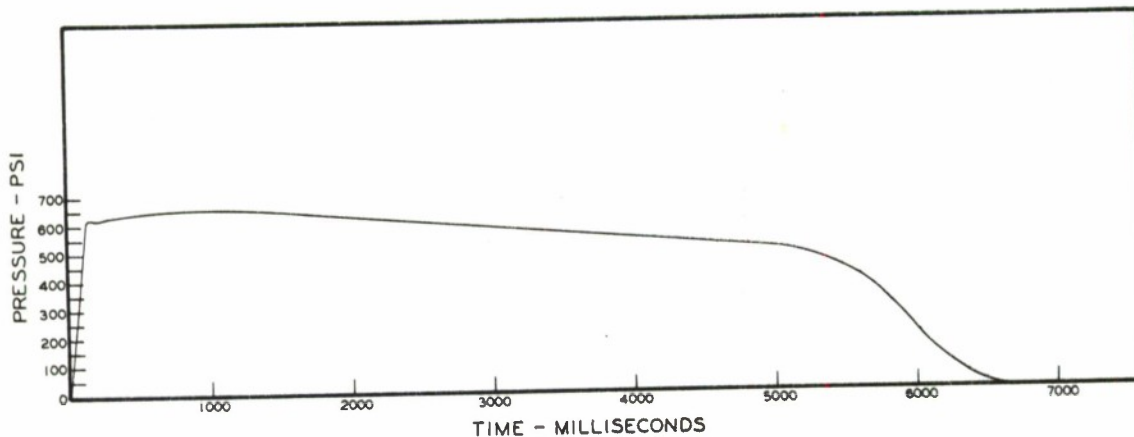


Fig. 1 Pressure time trace obtained from scale Pershing motor filled with petrin acrylate propellant.

PROCESSING OF PROPELLANTS TESTED FOR PERSHING MOTOR

To conserve time and expenditures in this program processing studies were limited to those necessary for the production of satisfactory test motors.

Because of the requirement for minimum burning rates unground ammonium perchlorate was used in all formulations tested. Unground perchlorate tends to settle much more rapidly than finer perchlorate, thus requiring some thickening of the propellant as cast. Tests in which settling was determined by examination of the tops and bottoms of six-inch test motors showed that an initial viscosity of 5000-6000 cp¹ was adequate to

¹Apparent Newtonian viscosity measured with a T spindle at 5 rpm with a Brookfield viscometer.

prevent appreciable settling if gel times were less than 3 hours. Viscosities of 3000-4000 cp (the normal range for QZ_{bn} propellant) permitted definite settling in the test motors. The mixing and casting facilities used were not entirely adequate for the casting of propellant having such a high viscosity in small motors¹ and the test motors contained a number of minor flaws. In spite of their rather poor appearance, most of the motors were fired for ballistic evaluation without blow-ups.

Small scale tests on compositions in which RDX replaced a part of the perchlorate showed that these compositions were very impact sensitive and none of the compositions were cast in 6-in. motors.

Several 6-in. test motors were cast with compositions in which a part of the perchlorate was replaced with ammonium nitrate. The desired low burning rates were obtained but impulse was also low. Facilities were quite inadequate for the processing of these compositions because humidity could not be controlled.

No further work on processing was attempted until a formula was specified for evaluation in larger motors (formula RM). Tests were then run to determine the polymerization characteristics of this propellant and to evaluate the physical properties. Considerable difficulty was encountered in controlling the gel time adequately for a large batch. A high inhibitor concentration was required (0.08% N-ndpa compared to 0.04 to 0.05% N-ndpa for QZ_{bn}) and polymerization rates were erratic. Suitable adjustment of the inhibitor concentration gave a degree of control that was adequate for processing RM composition. The normal monitoring technique used for large batches would probably permit satisfactory casting of 100-pound motors.

When processing was scaled up to the large mixer (100 gal) the

¹Six-inch diameter x 11.4-inch long with a 5-inch cylindrical perforation.

hold time required was too long and the first batch of RM propellant polymerized in the hold tank before it was cast. The inhibitor was increased in the second batch and casting seemed normal, but none of the propellant from this batch which was cast into motors cured.

The high concentration of flake aluminum used in the Pershing formula (RM) seemed to be largely responsible for the serious polymerization control problem. Before large batches could be successfully cast an alternative method of thickening the propellant mix would probably be required.

The physical properties of composition RM were not greatly different from those of QZ_{bn}, which has been fired successfully in 7000-pound motors. In fact, QZ_{bn} made with a reagent grade MgO, (used in the first 7000-pound motor fired) had a slightly lower tensile strength and the same elongation. Current production QZ_{bn} has a tensile strength about 25% higher than RM containing unground ammonium perchlorate.

TABLE II

Physical Properties of RM Propellant

<u>Specific Gravity</u>	<u>Physical Properties at 75° F¹</u>	
	<u>Tensile Strength</u> psi	<u>Elongation</u> %
1.78	41	30

¹Average of tests on samples from 10 batches.

Initial distribution of this report was made
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